

# Higher olefin hydroformylation in organic/aqueous biphasic system accelerated by double long-chain cationic surfactants

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## Abstract

The double long-chain cationic surfactants (DLCS) were used in higher olefin hydroformylation catalyzed by water-soluble rhodium catalyst in aqueous/organic biphasic system. The reaction rate was greatly accelerated and the value of TOF (turn over frequency defined as the moles of converted olefin per mole Rh per hour) in 1-dodecene hydroformylation could achieve  $2291 \text{ h}^{-1}$  even without stirring. When the reaction was carried out with stirring rate of 400 rpm, TOF increased to  $7472 \text{ h}^{-1}$ . These values of TOF were comparable with that in homogeneous catalysis system. The phenomenon is attributed to the formation of vesicle and the enrichment of rhodium catalyst in the interfacial layer, as well as the high transfer rate of olefins from the organic phase to the interface of vesicle. The acceleration mechanism of DLCS in biphasic catalysis was discussed. © 2006 Elsevier B.V. All rights reserved.

**Keywords:** Hydroformylation; Rhodium complex; Surfactant; Long-chain olefin; Biphasic catalytic system

## 1. Introduction

A persisting problem in higher olefin hydroformylation is the recovery and reuse of the expensive rhodium catalyst. The aqueous/organic biphasic catalysis offers a feasible way to solve the problem of separation of products from catalysts. Moreover it is economical and environmentally benign. However, owing to very low solubility of higher olefin in the aqueous phase and the higher energy barrier of olefin transfer from the organic phase to the aqueous phase the reaction rate is extremely low. Extensive efforts, such as adding co-solvent [1], co-ligand [2], amphiphilic phosphines [3–5], modified cyclodextrin [6–10] and surfactants [11–17] into the reaction solution, have been devoted to accelerate the reaction, but the reaction rate is still much lower under vigorous stirring conditions than that in homogeneous catalysis. Among the published reports, the accelerating effect of single long-chain cationic surfactants [9,18] and the multiple long-chain amphiphilic phosphines [5] are the most notable. Both the micelle formation in the former case and the vesicle formation in the latter case play an important role in promoting

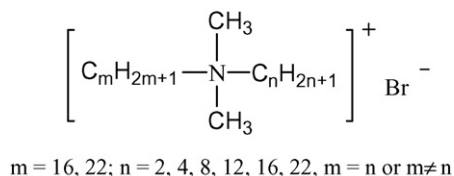
the reaction. In order to further improve the reaction rate of higher olefin hydroformylation, we synthesized a series of double long-chain cationic surfactants (DLCS) and used them in the biphasic catalytic reaction as promoter. Extremely fast rates of hydroformylation were attained in the presence of DLCS even without stirring. Here we report the highly active catalyst system and the acceleration mechanism of vesicle formed from DLCS in the solution.

## 2. Experimental

### 2.1. Materials

The sodium salt of tris(3-sulfonatophenyl)phosphine (TPPTS) and the catalyst precursor  $\text{RhCl}(\text{CO})(\text{TPPTS})_2$  were prepared according to the literature [19]. 1-Decene (Sigma, 96%), 1-dodecene (Across, 93–95%), 1-tetradecene (Sigma, 99%), 1-hexadecene (Fluka, 99%), 1-octadecene (Sigma, 97%), 1-*N,N*-dimethylhexadecyl amine (C. P.), *N,N*-dimethyl docosylamine (C. P.), bromoethane (C. P.), *n*-butyl bromide (C. P.), *n*-octyl bromide (C. P.), *n*-dodecyl bromide (C. P.), cetyltrimethyl ammonium bromide (CTAB) (C. P.) and *n*-hexadecyl bromide (C. P.) were commercially available and used as received without further purification. Hydrogen (99.99%) and carbon monoxide (99.9%) were mixed directly at

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Scheme 1. The structure of cationic double-chain surfactants.

the molar ratio of 1:1. Distilled and deionized water was used in all experiments. A series of DLCS (Scheme 1) was synthesized by refluxing dimethylalkyl amine and *n*-alkyl bromide in acetone or alcohol solution as described in the literature [20].

## 2.2. Hydroformylation and catalytic recycle test

RhCl(CO)(TPPTS)<sub>2</sub>, TPPTS, DLCS, and distilled and deionized water were added into a stainless steel autoclave with a magnetic stirrer, and the aqueous solution was stirred for 5 min at room temperature. After the stirring was stopped, olefin was carefully added using a transfer pipette along the autoclave wall, and then syngas (H<sub>2</sub>:CO = 1:1) was introduced. The hydroformylation was carried out under the desired pressure and temperature with stirring or without stirring in the entire reaction process. When the reaction was completed, the reaction mixture was transferred to a tube and separated into three layers with centrifugation. The upper organic layer was carefully taken out from the top of the tube and analyzed by GC HP1890II with a FID and a capillary column of SE-30, 30 m × 0.25 mm I.D. In the case of catalyst recycles, both the bottom and interfacial layers were reused in the next run with the addition of fresh olefin.

## 2.3. Determination of rhodium contents in solution

The organic layer, interfacial emulsion layer and aqueous layer were separated carefully and each was concentrated under reduced pressure for removing the volatile compounds. The

remainder was treated by aqua regia under refluxing conditions for a long time to ensure that all of the organic compounds were completely carbonized and that the majority of aqua regia in the solution was removed by heating. The distilled water was introduced into the remaining solution and the carbide was separated by filtration. The rhodium contents in the aqueous solution were determined by ICP on an IRIS Advantage Instrument.

## 2.4. TEM observation of vesicle image of [(C<sub>16</sub>H<sub>33</sub>)<sub>2</sub>N(CH<sub>3</sub>)<sub>2</sub>]Br

Ultrasonication was employed in the preparation of aqueous solution of [(C<sub>16</sub>H<sub>33</sub>)<sub>2</sub>N(CH<sub>3</sub>)<sub>2</sub>]Br, as well as the mixed solution containing surfactant, 1-dodecene, TPPTS and catalyst RhCl(CO)(TPPTS)<sub>2</sub> for Transmission Electron Microscope (TEM) observations. Negative staining was used in the observation of the vesicle image with a JEM-200CX TEM operating at 120 keV. A 230 mesh copper grid coated with carbon formvar was soaked in the surfactant solution for about 60 min. The grid was drained on absorbent tissues, then the grid was stained with a drop of a 1% (w/v) solution of uranyl acetate. After 10 min, the liquid drop was removed with filter paper and the resulting stained sample was observed.

## 3. Results and discussions

### 3.1. Acceleration effect of DLCS

The hydroformylation of 1-dodecene was performed in a stainless steel autoclave under the conditions as described in Table 1. The reaction was very slow in the presence of single long-chain surfactant CTAB without stirring. However, the reaction was dramatically accelerated by a factor of 11–31 in the presence of DLCS. The influence of alkyl chain length in DLCS on the reaction rate indicated that when one alkyl chain length was fixed, for example at C<sub>22</sub>H<sub>45</sub>, TOF (turnover frequency defined as moles of converted olefin per mole Rh per

Table 1  
Effect of different surfactants on 1-dodecene hydroformylation without stirring

Entry	Surfactant	Conversion (%) <sup>a</sup>	S <sub>ald</sub> (%) <sup>b</sup>	S <sub>alk</sub> (%) <sup>c</sup>	S <sub>iso</sub> (%) <sup>d</sup>	L/B <sup>e</sup>	TOF (h <sup>-1</sup> ) <sup>f</sup>
1	C <sub>22</sub> H <sub>45</sub> N(CH <sub>3</sub> ) <sub>2</sub> C <sub>16</sub> H <sub>33</sub> Br	91.6	83.0	11	6.0	3.1	1073
2	C <sub>22</sub> H <sub>45</sub> N(CH <sub>3</sub> ) <sub>2</sub> C <sub>12</sub> H <sub>25</sub> Br	91.9	85.2	9.2	5.6	3.5	1077
3	C <sub>22</sub> H <sub>45</sub> N(CH <sub>3</sub> ) <sub>2</sub> C <sub>8</sub> H <sub>17</sub> Br	90.1	88.5	7.4	4.1	3.9	1056
4	C <sub>22</sub> H <sub>45</sub> N(CH <sub>3</sub> ) <sub>2</sub> C <sub>4</sub> H <sub>9</sub> Br	34.1	56.3	28.8	14.9	3.6	399
5	C <sub>22</sub> H <sub>45</sub> N(CH <sub>3</sub> ) <sub>2</sub> C <sub>2</sub> H <sub>5</sub> Br	12.9	60.9	23.8	15.2	3.9	151
6	C <sub>16</sub> H <sub>33</sub> N(CH <sub>3</sub> ) <sub>2</sub> C <sub>16</sub> H <sub>33</sub> Br	94.8	83.3	10.8	5.9	2.5	1111
7	C <sub>16</sub> H <sub>33</sub> N(CH <sub>3</sub> ) <sub>2</sub> C <sub>12</sub> H <sub>25</sub> Br	82.9	77.5	14.0	8.5	3.2	971
8	C <sub>16</sub> H <sub>33</sub> N(CH <sub>3</sub> ) <sub>2</sub> C <sub>8</sub> H <sub>17</sub> Br	38.2	73.6	16.1	10.3	3.5	448
9	CTAB	3.0	–	–	–	3.0	35.1

Reaction conditions: 1-dodecene: 2 mL (9.0 mmol), [RhCl(CO)(TPPTS)<sub>2</sub>]: 9.6 × 10<sup>-4</sup> mol/L, [TPPTS]/[Rh] = 18, H<sub>2</sub>O: 4.0 mL, syngas pressure: 2.0 MPa (constant), 100 °C, 2 h, surfactant concentration = 5.0 mmol/L.

<sup>a</sup> Molar conversion of olefin.

<sup>b</sup> Selectivity of aldehydes.

<sup>c</sup> Selectivity of *n*-dodecane.

<sup>d</sup> Selectivity of isomeric olefins.

<sup>e</sup> Molar ratio of linear to branched aldehyde.

<sup>f</sup> Turnover frequency defined as the moles of converted olefin per mole Rh per hour.

Table 2  
Effect of different surfactants on hydroformylation of 1-dodecene with stirring

Entry	Surfactant	Conversion (%)	$S_{ald}$ (%)	$S_{alk}$ (%)	$S_{iso}$ (%)	L/B	TOF ( $h^{-1}$ )
10	$C_{22}H_{45}N(CH_3)_2C_{16}H_{33}Br$	83.3	96.9	2.6	0.5	3.7	1951.7
11	$C_{22}H_{45}N(CH_3)_2C_{12}H_{25}Br$	86.2	91.7	5.4	2.9	3.3	2019.7
12	$C_{22}H_{45}N(CH_3)_2C_8H_{17}Br$	85.9	93.0	3.4	3.6	3.6	2012.6
13	$C_{16}H_{33}N(CH_3)_2C_{16}H_{33}Br$	90.7	91.4	3.4	5.2	3.1	2125.1
14	$C_{16}H_{33}N(CH_3)_2C_{12}H_{25}Br$	94.5	93.0	4.2	2.8	3.3	2214.1
15	CTAB	20.9	80.4	10.0	9.6	2.8	489.7

Reaction conditions: reaction time is 1 h, stirring rate is 400 rpm, the others are the same as in Table 1.

hour) rose with the increase in the length of another alkyl chain (Table 1, entries 1–5). It was worth noting that when  $n = 8$  in  $(C_{22}H_{45})N(CH_3)_2(C_nH_{2n+1})Br$ , TOF took a jump from  $399 h^{-1}$  ( $n = 4$ ) to  $1056 h^{-1}$  ( $n = 8$ ) (Table 1, entries 3 and 4). Similar phenomena were also observed in the series of  $(C_{16}H_{33})N(CH_3)_2(C_nH_{2n+1})Br$ , but the jump occurred at  $n = 12$  (Table 1, entries 7 and 8) wherein TOF increased from  $448 h^{-1}$  ( $n = 8$ ) to  $971 h^{-1}$  ( $n = 12$ ). This trend suggests that when two alkyl chains of surfactants are long enough, the chains act synergistically to accelerate the reaction rate. The increased TOF of  $2291 h^{-1}$  was observed in the presence of  $(C_{16}H_{33})_2N(CH_3)_2Br$  when the molar ratio of 1-dodecene to rhodium was increased to 3516 and the reaction time was shortened to 1 h. The TOF was comparable with the TOF of  $2343 h^{-1}$  in homogeneous hydroformylation of 1-dodecene catalyzed by  $HRh(CO)(PPh_3)_3/PPh_3$  in toluene solution under the same reaction conditions.

Table 1 shows that the selectivity for aldehyde is not satisfactory when the reaction is carried out without stirring. The lower selectivity for aldehyde is owing to the enhancement of both hydrogenation and isomerization of olefin. It is possible that the transfer rate of CO in the solution is much lower than that of  $H_2$  without stirring in the reaction process, because the diffusion coefficient of  $H_2$  is much high than that of CO, and thus hydrogen could easily approach rhodium complexes to form semihydrogenation intermediate, which would be favorable for the hydrogenation and isomerization of olefin. When the reaction is carried out at a stirring rate of 400 rpm, the transfer rates of all reactant molecules are significantly accelerated, the values of TOF substantially increase and the selectivities for aldehyde also obviously rise. The data are shown in Table 2. When the mole ratio of 1-dodecene/Rh is increased to 4686 and the reaction time is shortened to 0.5 h, the TOF achieves  $7472 h^{-1}$  and the selectivity for aldehyde increases to 93.9%, which are also comparable with the TOF ( $7865 h^{-1}$ ) and the selectivity (96.3%) in homogeneous hydroformylation of 1-dodecene. The results further confirm that the extremely high rate of olefin hydroformylation in the biphasic catalytic system can be achieved in the presence of double long-chain cationic surfactants.

### 3.2. Effect of olefin chain length and TPPTS/Rh ratio

The acceleration effect of DLCS has been also observed in the hydroformylation of different long-chain olefins as shown in Fig. 1. The results indicate some differences among different olefins. For example, when the surfactant  $(C_{16}H_{33})_2N(CH_3)_2Br$

is used for biphasic reactions the TOF for 1-octene is  $1157.2 h^{-1}$ , while the TOF for 1-hexadecene decreases to  $722.1 h^{-1}$ . The decrease in the olefin conversion rate with the increase in its chain length is due to the reduction of its reactivity, but the conversion rates are still fast without stirring even for 1-octadecene which is insoluble in water.

The acceleration effect of DLCS is influenced by the mole ratio of TPPTS/Rh. When the mole ratio of TPPTS/Rh is 18 (Table 3, entry 16), the conversion of 1-dodecene and TOF are 94.8% and  $1111 h^{-1}$ , respectively, within 2 h. Increasing the mole ratio of TPPTS/Rh, the conversion of 1-dodecene decreases gradually. When the ratio of TPPTS/Rh is 45 (Table 3, entry 19), 1-dodecene conversion and TOF are 59.4% and  $696.1 h^{-1}$ , respectively. Although the higher TPPTS/Rh ratio exhibits a negative effect on the reaction acceleration, it produces a positive effect on the selectivity to aldehyde.

### 3.3. Catalytic recycle test

The catalyst recycling in 1-dodecene hydroformylation was carried out in the presence of  $(C_{16}H_{33})_2N(CH_3)_2Br$  under the conditions listed in Table 4. After the reaction, the mixed solution displays three layers as shown in Fig. 2: upper organic layer (1.9 mL) is colorless and transparent, interfacial emul-

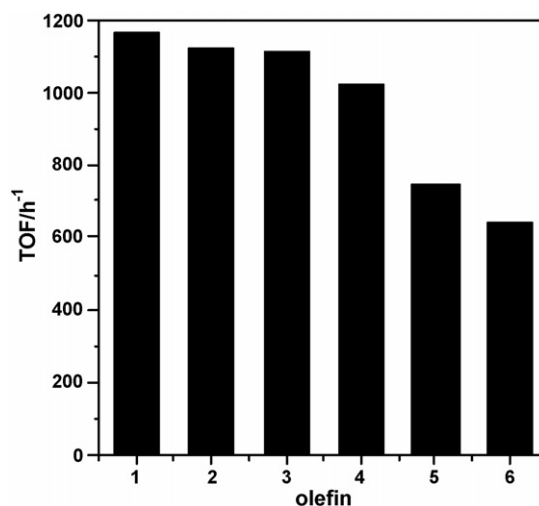


Fig. 1. Hydroformylation of  $\alpha$ -olefin of different chain length without stirring in the presence of  $(C_{16}H_{33})_2N(CH_3)_2Br$ . Reaction conditions are the same as in Table 1. (1) 1-octene; (2) 1-decene; (3) 1-dodecene; (4) 1-tetradecene; (5) 1-hexadecene; (6) 1-octadecene.

Table 3  
Effect of mole ratio of TPPTS/Rh on hydroformylation of 1-dodecene

Entry	TPPTS/Rh (mole ratio)	Conversion (%)	$S_{ald}$ (%)	$S_{alk}$ (%)	$S_{iso}$ (%)	$L/B$	TOF ( $h^{-1}$ )
16	18	94.8	83.3	10.8	5.9	2.5	1111
17	25	81.5	75.6	16.6	7.8	3.4	955.1
18	35	68.9	90.4	6.0	3.6	2.5	807.4
19	45	59.4	94.9	3.9	1.2	3.0	696.1

Reaction conditions:  $(C_{16}H_{33})_2N(CH_3)_2Br$  is 5.0 mmol/L, the others are the same as in Table 1.

Table 4  
Catalyst recycle tests

Entry	Run	Conversion (%)	$S_{ald}$ (%)	$S_{alk}$ (%)	$S_{iso}$ (%)	$L/B$	TOF ( $h^{-1}$ )
20	1	97.9	92.8	2.2	5.0	3.4	532.2
21	2	91.3	78.6	15.2	6.2	3.0	420.4
22	3	90.9	72.8	19.6	7.6	2.9	387.7
23	4	90.8	78.9	15.2	5.9	3.1	419.8
24	5	92.1	77.6	15.1	7.3	3.2	418.8
25	6	91.3	82.3	12.5	5.2	3.2	440.3
26	7	86.5	71.8	20.3	7.9	3.1	363.9
27	8	87.9	67.3	23	9.7	3.0	346.6

Reaction conditions:  $(C_{16}H_{33})_2N(CH_3)_2Br$  is 5.0 mmol/L,  $[TPPTS]/[Rh]=35$ , the others are the same as in Table 1, except that the reaction time is 4 h.

sion layer (0.4 mL) is brown and the bottom aqueous layer (3.7 mL) is yellow. The rhodium contents in three layers have been determined by Inductively Coupled Plasma–Atomic Emission Spectrometry (ICP–AES) and the distributions of rhodium are: 1.0% (organic layer), 29.2% (middle layer) and 69.8% (aqueous layer), respectively. Although most of rhodium catalyst still remains in the aqueous phase, the rhodium concentration in the middle interfacial layer is 70.3 ppm, which is much higher than 18.1 ppm in the aqueous layer and 0.52 ppm in the organic layer. Therefore, the leaching of rhodium into the organic phase is extremely low. This is further confirmed by the data of catalytic recycle.

The recycle experiments using the solution containing both the middle interfacial layer and the aqueous layer in subsequent reactions show that the activity does not obviously decrease. If only the aqueous phase is used for the subsequent reaction, 1-dodecene conversion decreases significantly from 90% to 62%.

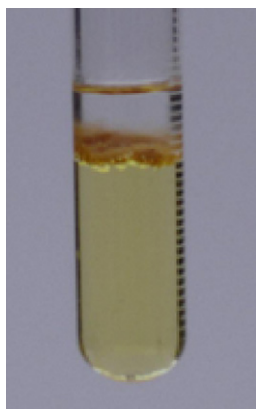


Fig. 2. The picture of the three layers after the reaction.

### 3.4. Acceleration mechanism of DLCS

The great acceleration effect of the DLCS is attributed to the formation of vesicle constructed from bilayer membrane of DLCS in aqueous solution. The bilayer structure is associated with the strong hydrophobic effect of two long alkyl chains in surfactants. The vesicles are observed by TEM in  $(C_{16}H_{33})_2N(CH_3)_2Br$  aqueous solution and the solution containing  $(C_{16}H_{33})_2N(CH_3)_2Br$ , 1-dodecene, TPPTS and  $RhCl(CO)(TPPTS)_2$  (Figs. 3 and 4), but no vesicles are found in the solution containing CTAB.

According to the study of vesicle structure in the solution of double long-chain surfactants, there are internal and external aqueous phases in the vesicles [21]. This structure significantly increases biphasic interface area and thereby rhodium catalyst species with negative charge are easily absorbed on the vesicle hydrophilic surface with positive charge. The situation is favorable for enrichment of rhodium catalysts on the hydrophilic

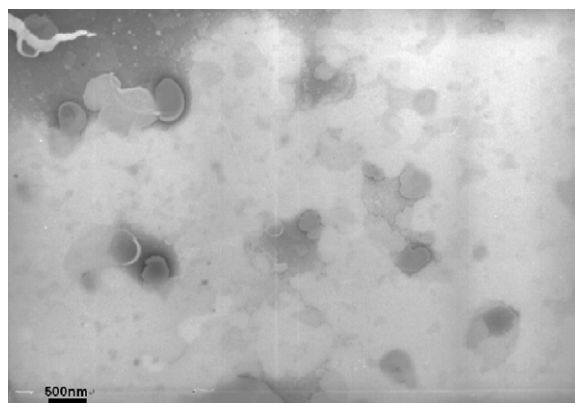


Fig. 3. TEM image of  $(C_{16}H_{33})_2N(CH_3)_2Br$  solution.  $[(C_{16}H_{33})_2N(CH_3)_2Br]$ : 4.0 mmol/L.

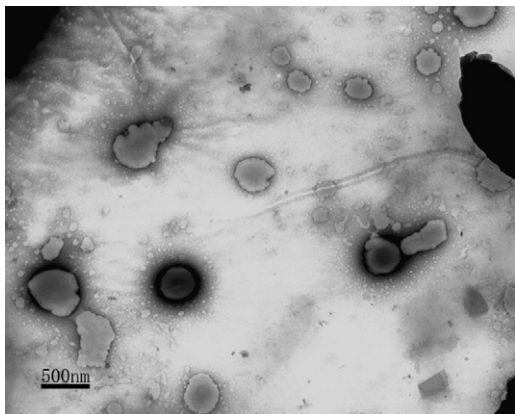


Fig. 4. TEM image of the mixed solution containing TPPTS,  $\text{RhCl}(\text{CO})\text{-(TPPTS)}_2$ , 1-dodecene,  $\text{H}_2\text{O}$  and  $(\text{C}_{16}\text{H}_{33})_2\text{N}(\text{CH}_3)_2\text{Br}$ .  $[(\text{C}_{16}\text{H}_{33})_2\text{N}(\text{CH}_3)_2\text{Br}]$ : 4.0 mmol/L;  $[\text{TPPTS}]$ : 17.3 mmol/L;  $[\text{RhCl}(\text{CO})(\text{TPPTS})_2]$ : 0.96 mmol/L; 1-dodecene/ $\text{H}_2\text{O}$ : 4/10 (v/v).

surface of vesicle. At the same time, the bulk olefins are solubilized in the hydrophobic interior of bilayer structure formed from two long alkyl chains of surfactant and thus the local concentration of olefin in vesicle is extremely high [5]. In this microcircumstance the distance of 1-dodecene from the interior of vesicle to interface is shortened, and moreover mass transfer energy barrier of olefins across the interface between organic and aqueous phases is considerably reduced owing to the influence of interfacial electrostatic field of vesicle. The combination of the above factors would create a very advantageous condition for the coordination of olefin with rhodium catalytic species. In the biphasic system every vesicle can be considered as a microreactor in which both the catalyst and substrates are highly concentrated, and consequently the reaction rapidly occurs.

#### 4. Conclusion

The investigation demonstrated that the biphasic catalytic system containing double long-chain cationic surfactants as promoter is very effective for hydroformylation of higher olefins. In this biphasic system the reaction rate can near the same level as that in homogeneous catalysis. This discovery could be applied to other biphasic catalytic reactions and the biomimical catalysis.

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